UNITED STATES DEPARTMENT OF AGRICULTURE

AGRICULTURAL RESEARCH SERVICE

SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

C I T R U S

P R O C E S S I N G C O N F E R E N C E

U. S. Fruit and Vegetable Products Laboratory

Winter Haven, Florida

September 20, 1961

PROGRAM AND ABSTRACTS OF PAPERS

ELEVENTH CITRUS PROCESSING CONFERENCE

September 20, 1961

Florida Room, Citrus Building
Winter Haven, Florida

ORGANIZATIONS PARTICIPATING

IN

ELEVENTH CITRUS PROCESSING CONFERENCE

SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Florida
U. S. Fruit and Vegetable Products Laboratory, Weslaco, Texas

WESTERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

Fruit and Vegetable Chemistry Laboratory, Pasadena, California
Western Regional Research Laboratory, Albany, California

PROGRAM

CITRUS PROCESSING CONFERENCE
September 20, 1961

MORNING SESSION - 9:45 A. M.

(M. K. Veldhuis, In Charge, U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Florida, Presiding)

AFTERNOON SESSION 1:30 P. M.

ADDITIONAL CONTENTS

RAPID ESTIMATION OF d-LIMONENE IN CITRUS JUICES

M. K. VELDHUIS and G. L. K. HUNTER
U. S. Fruit and Vegetable Products Laboratory
Winter Haven, Florida

The Clevenger method is the standard procedure for estimating recoverable oil in citrus juice products, and with care it will give good results. It requires about 90 minutes to complete the analysis. This method is satisfactory for establishing grade or checking a pack already prepared, but it leaves much to be desired as a quality control method because it is so slow.

Dr. R. B. McKinnis (1) has modified the procedure so that only 500 ml. of sample is needed instead of the 2000 ml. for the Clevenger method. The smaller bore in the measuring burette required the addition of a detergent so that the oil would wet the bore. The distillation can be completed in 30 minutes. For one reason or another this method has not been widely accepted.

Dr. E. R. Burdick (2) introduced the idea of adding acetone to the orange juice so as to facilitate the release of the oil and distilling the acetone and volatile oils together. When water was added to the distillate an emulsion formed and the amount of oil present was judged by density with a colorimeter. This reduced the time to about 10 minutes. Kilburn (3) further modified this method by substituting isopropanol for acetone which permitted the analysis of juices with lower oil content such as those prepared for babies. This procedure is in use in some laboratories as a control method.

The current investigation was undertaken to further speed the procedure by direct steam injection and to quantitatively determine oil present colorimetrically by the addition of bromine water to the distillate. It was proposed that live steam be used to speed the distillation and that this be combined with the Burdick or Kilburn method.

A pressure reducer and steam trap were arranged to yield dry steam at about 5 psi. A Kjeldahl still was modified so that live steam was injected by inserting a 1/4 inch stainless steel tube through the stopper leading to the bottom of the flask. A 50-inch length of 3/8 inch stainless steel tube was coiled into a compact condenser. By this arrangement it was found that a 100 ml. sample could be brought to a boil in 25 seconds and distillation could continue at a rate of 25 ml. per minute. At higher rates the Kjeldahl trap flooded excessively. A bypass valve was used to bring the sample to a boil rapidly; the valve was then closed allowing a pre-set needle valve to control the rate of distillation.

With the addition of acetone or isopropanol to the juice, an oil-in-water emulsion was obtained. Some difficulty was experienced in obtaining consistent direct colorimetric readings on this emulsion; nevertheless, this provides a very rapid method which may be suited to many control purposes as the distillation can be completed in about 3 minutes.

Another approach has been made toward a more quantitative method by brominating the distillate with bromine water and measuring the residual bromine by means of a colorimeter. The principal constituent of peel oil is d-limonene which has two double bonds that react with bromine. Isopropanol cannot be used because it also absorbs bromine; however, acetone has been found to be suitable as it reduces bromine only slowly. This procedure is both rapid and precise.

In the tentative procedure a sample containing about 0.01 ml. of d-limonene is diluted to 100 ml. and placed in a short-necked 300 ml. Kjeldahl flask with 10 ml. of reagent grade acetone. Steam is applied as previously described and 60 ml. distilled into a 100 ml. glass stoppered graduate. Twenty milliliters of a diluted solution (15 to 1) of saturated bromine water is rapidly added to the contents of the graduate. The graduate is shaken thoroughly and a portion poured into a dry colorimeter tube. The colorimeter is read with a 420 mμ (blue) filter without delay. The reading is compared with a standard curve prepared in a similar manner with known mixtures of either peel oil-acetone-water, or d-limonene-acetone-water, or by comparison with samples analyzed by the Clevenger method. The entire procedure can be completed in about 4 minutes and gives more reproducible results than measurement of the optical density of the oil-acetone-water emulsions.

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RECENT ADVANCES IN THE CHEMISTRY OF THE CITRUS FLAVONOIDS

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Work has continued on the nature of the sugar constituents present in the flavonoid glycosides of citrus fruits. The types of glycosides now known to occur are the following: - the rhamnoglucosides (including both rutinosides and neohesperidosides); simple glucosides; and carbon-carbon linked glycosides. Some recently found examples of these will be discussed.

The flavonols limocitrin and limocitrol (both from lemons) have been mentioned in previous reports. These compounds are 3,5,7,4'-tetrahydroxy-8,3'-dimethoxyflavone and 3,5,7,4'-tetrahydroxy-6,8,3'-trimethoxyflavone, respectively. A third new flavonol from lemons, isolimocitrol, has been found recently and its structure shown by means of ultraviolet spectra. It appears to be 3,5,7,3'-tetrahydroxy-6,8,4'-trimethoxyflavone. Limocitrin, limocitrol and isolimocitrol have all been isolated in the form of their glucosides, which have been found to be, in each case, the 3-beta-D-glucoside. The chemical and spectral evidence for these structures will be discussed.

Study of the flavonoid glycosides of oranges has revealed the occurrence of naringenin 7-rhamnoglucoside and isosakuranetin 7-rhamnoglucoside. As neither of these compounds is bitter, it may be inferred that the sugars are in the form of rutinose rather than neohesperidose. (The corresponding neohesperidose derivatives, which are found in grapefruit, are the bitter flavanones naringin and poncirin). A summary of the available information on the occurrence of neohesperidose and rutinose derivatives in citrus will be given.

The carbon-carbon glycosides of flavonoid compounds have been shown to occur in several plant species but never in citrus. Vitexin, the most thoroughly studied of these compounds, has long been known as a constituent of the tree Vitex lucens. However, its structure is not known with complete certainty. Recently, we have isolated vitexin in small yield from Valencia oranges and have also obtained several similar C-C glycosides of flavones from other citrus fruits.

A number of derivatives of various rutinose and neohesperidose glycosides have been prepared in order to study the relations between chemical constitution and taste in these compounds. These data will be discussed briefly.

THE EFFECT OF PULP IN ORANGE JUICE ON HYDROMETER VALUES

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The Florida Citrus Commission and other interested groups in the citrus industry requested that the U. S. Department of Agriculture undertake a thorough review of the official inspection procedures used in Florida for sampling and testing oranges for processing to determine pounds of solids per box. In accordance with this request a special survey committee, composed of U. S. D. A. and Florida Experiment Station personnel, was formed. Following its survey of sampling and testing procedures currently in use, the Committee recommended that certain phases of these procedures be subjected to critical study and evaluated as possible sources of error. One of the studies suggested was to determine if variations in pulp content affect the Brix reading of orange juice, and the U. S. Fruit and Vegetable Products Laboratory was asked to conduct the investigation.

Thirty-one samples of Valencia orange juice were examined for the effect of contained pulp on their hydrometer Brix values. Twenty-five of these were brought to the Laboratory directly from State testing stations at concentrate plants where FMC extractors were used, each sample being prepared from a separate load of fruit. A few of these contained so much pulp that the hydrometer could not be used until some of the pulp had been strained out, and all contained more pulp than is common in retail products. Six other samples were prepared from separate lots of fruit with a Brown extractor and finished through an 0.040" finisher screen, three at 7 lbs. and three at 10 lbs. pneumatic pressure on the finisher shaft.

Hydrometer values were obtained on each sample with four levels of pulp content: full pulp, with 1/4 of the pulp removed by straining through cheesecloth, with 5/8 of the pulp removed, and with all the pulp removed. This was accomplished by straining 1/4 of the juice and mixing it with the unstrained, then straining 1/2 the mixed juice, and finally straining all the juice. Cloths were wet with an extra portion of the juice being tested and tightly squeezed before their tares were taken, and similar squeezing pressures were applied to "dry" the cloths with and without recovered pulp. Total pulp was calculated by adding the weights of pulp recovered at each straining. Pulp contents ranged from 0.65% to 4.33% by weight. In order to eliminate an effect from air bubbles, about 800 ml. of the prepared samples were deaerated and carefully remixed before taking the hydrometer readings. All readings were taken with the same hydrometer with enclosed thermometer, and were corrected to its calibration temperature, 20° C. Readings were made to the nearest 0.05° Brix. Hydrometer values of strained juice ranged from 9.79° to 13.41° Brix.

Differences due to incremental removal of pulp were small and variable. Of 93 differences, however, only 8 were increases. Average reductions in hydrometer values for each step in pulp removal were successively 0.040°, 0.044°, and 0.040° Brix, with an average over-all reduction of 0.124° Brix. The average reduction caused by the removal of 1% pulp was 0.051° Brix.

PROGRESS REPORT ON THE PREPARATION OF PULP FORTIFIED CONCENTRATE FROM RUBY RED GRAPEFRUIT

BRUCE J. LIME
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Weslaco, Texas

The U. S. Fruit and Vegetable Products Laboratory at Weslaco, Texas, has been working for several years on the utilization of red grapefruit. One of the difficulties encountered in processing is the color of the juice products. When processed in the same manner as white grapefruit the juice has too much color to pass the standards for white juice and does not have enough color to pass the standards for red grapefruit juice. Previous studies have indicated that the color of single strength juice from Ruby Red grapefruit can be enhanced by pulp fortification. This study was undertaken to determine what stabilization measures, if any, were necessary when the pulp fortification procedures were applied to the production of frozen concentrate produced from Ruby Red grapefruit juice. Eighteen concentrate packs were prepared with varying degrees of heat stabilization of the raw juice and the high pulp cutback juice. Heat treatment by flash pasteurization at temperatures of 170°, 175°, 180° and 185° F. were used. The packs were frozen and kept in storage at -10° F. until analyzed.

Preliminary analyses of the concentrate packs after six months storage have indicated that the pulp fortified cutback improved the color of the concentrates. All heat treatments improved the stability of the cloud of the concentrate. Heat treatments of 180° and 185° F. were rated lower in flavor than heat tretments of 170° and 175° F.

FOAM-MAT DRYING OF ORANGE JUICE: PROGRESS IN FOAM PREPARATION, DRYING, AND STORAGE STUDIES[1/]

OWEN W. BISSETT, JAMES H. TATUM, and CHARLES J. WAGNER, JR.
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Winter Haven, Florida

Foam-mat drying as discussed at our Conference last year was concerned primarily with the equipment and its use in the preparation and drying of foamed concentrates. The meeting closed with a successful demonstration of the process at the Laboratory. In principle, "foam-mat" drying depends upon addition of a small amount of a stabilizer to orange concentrate and the generation of a foam by beating in a food mixer using a wire whip. The foam is passed through the drier as small strands on a Teflon-fibreglas belt. Drying is accomplished by hot air blown across the belt and the dried material removed in a low humidity packaging room.

The principal phases of the process investigated during the past year include foam preparation, drying studies, packaging, and storage. These will be discussed in the order listed.

Foam stabilizers must permit the formation of a foam which can be handled in the feeders without breaking down. The foam must also retain its shape on the drying belt and give a satisfactory reconstituted product. The product used in most previous studies was a monoglyceride from hydrogenated lard. Most of the additional stabilizers tried were discarded because the foam was not sufficiently stiff or degraded rapidly during handling or drying. A product designated as a glyceryl monostearate has shown promise. Another type of product, a modified soya-bean protein, has shown the most promise when used with about one-fourth its weight of methyl cellulose. The latter has a distinct advantage of yielding a more natural color in the reconstituted product and reconstitutes more readily. The density of the foam is somewhat greater and permits a heavier belt loading rate than monoglyceride, but monoglyceride foamd dry in a shorter time so drier capacity remains the same. Neither of these two stabilizers contributed appreciably to flavor.

Drying studies have been made seeking to determine the effects of different drying schedules on the moisture content and flavor of the products. Modifications of the equipment have greatly increased theaccuracy of control of belt loading rate, and of air temperatures and air speeds. The drier is divided into four sections with each under separate temperature control.

1/ Cooperative investigations of the Florida Citrus Commission and the Southern and Western Utilization Research and Development Divisions.

It is thus possible to use air at 200° to 220° F. in the first section where moisture content is comparatively high and evaporation limits product temperature. Subsequent sections are controlled at lower temperatures depending on belt speed, loading rate, foam stabilizer used, and maximum desired product temperature.

A series of drying studies was conducted including use of both protein and monoglyceride stabilizers. Maximum foam temperatures during drying were 155°, 170°, and 185° F. These temperatures were maintained for periods of 2 to 10 minutes. Product moisture in the protein stabilized powders varied from 1.5% to 4% and in those containing the monoglyceride from 0.5% to 3%. All products dried at 185° F. had a definite scorched flavor. In products dried at lower temperatures another flavor was observed which has been associated with dried orange juice. It was present in varying degree depending on the drying conditions. At lower concentrations it was obscured by the addition of locked-in oil used for flavor fortification. These data indicate that 185° F. is too high a temperature for satisfactory flavor.

Preliminary studies of the effects of nitrogen gas and air packaging on flavor stability have been made. It had been thought that the presence of oxygen in the package would favor oxidative changes which would be observed as a flavor difference by the taste panel. Products were stored at -90°, -40°, 0°, and 70° F. and observed for flavor change for periods up to 8 weeks. No flavor differences were noted between powders packed in air or nitrogen. Additional studies are planned.

The flavor stability of "foam-mat" dried orange juice during storage at several temperatures and moisture contents is being investigated. Two orange juice powders, one containing a protein and the other a monoglyceride and having a moisture content of the order of 2.2% were packaged with a desiccant and stored at 70° and 85° F. The taste panel observed a flavor change in both products after 2 weeks in 85° F. storage. Stability was greater in 70° F. storage. At this temperature a difference was noted in products containing monoglyceride after 8 weeks and in those containing the protein after 16 weeks. Thus a greater stability is indicated for the protein stabilized materials even though moisture release is more rapid in products containing the monoglyceride.

The in-package desiccant is expensive, bothersome, and a sales hazard; and its elimination would be a distinct advantage. It is not feasible to reduce the moisture content during regular drying to less than about 2.25 percent at which moisture content powdered orange juice has limited storage life; hence, the in-package desiccant is needed to gradually decrease the moisture content in the can. A possible alternate procedure might be to reduce the moisture content to some lower figure by a secondary drying process before packaging. Powders of low moisture content have been produced by maintaining the product at low humidity under vacuum over a period of time at 70° F. Powders with moisture contents of 1.37%, 1.24%, and 0.92% have been prepared and additional lots with lower moisture contents are to follow. Storage life at 70° and 85° F. will be determined. Several methods of rapidly reducing powdered orange juice to low levels are being tried.

CONTINUOUS FOAM-MAT DRYING

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Foam-mat drying is a new way of producing instant food powders by air drying stable foams. Foams can be made by adding a small proportion of an edible foam stabilizer. One such stabilizer is a combination of solubilized soya protein and methyl cellulose. In various proportions and types, these two edible substances can be used to stabilize a wide variety of fruit and vegetable purees and concentrates, including those of the various citrus fruits.

In order to dry these foams quickly, they must be spread out in such a way that they are in good contact with the drying air. Air flowing through a thin air-punctured mat of foam lying on perforated plate or tray is one effective way of getting good contact. In order to use the air repeatedly and efficiently, a deep bed may be formed by stacking these trays up in the duct through which the air flows. A good arrangement involves a concurrent flow of fast hot air followed by a countercurrent flow of decreasing velocity and temperature. This has been done by continuously raising the trays through the duct, removing them dry at the top. Hot air flows into the bottom of the stack through the fresher trays and out at a central exit. Warm air enters the top of the stack and flows through the oldest trays. Some of the hot air enters the stack midway in this countercurrent section joining the warm air. The mixture flows through the remaining trays of the countercurrent section, leaving the stack at the same exit as the spent concurrent air from below.

Several pilot plant dryers of this type have been built, including one at WRRL. The WRRL unit is arranged for continuous and automatic operation beginning with food at one end and yielding dry powder at the other end. A continuous foaming device meters food stabilizer and gas together, produces a foam, and delivers it to the feeder. This unit extrudes the foam onto perforated trays and punctures or craters the mat. The trays then move through the dryer proper. Filtered hot air is delivered to the dryer by the blower unit. The dry material is conveyed to the detraying unit which is located in a low humidity room. The detrayer plows off the product and brushes the trays clean for reuse. The powder is filled into bags by gravity, while being flushed with nitrogen. The Albany unit can produce about 20 lbs. of orange juice powder per hour.

It is best to pack fruit powders as dry as possible in order to retard spoilage or caking. It seems difficult to get foam-mat dried products below 2.5% moisture without scorching them. We therefore have added small amounts of various solvents to these powders to facilitate final drying. A weight of a very hydrophilic solvent equal to 10% of the powder may be sprayed onto the powder without clumping it. This solvent,

together with most of the remaining water, can easily and completely be removed at room temperature in vacuum or a gentle air stream. Ethanol is a good choice for the purpose, although methanol is even faster. This technique is also usable for removing residual moisture from freeze dried food pieces. We suggest the term "Extractive Drying" for this method. We hope that in-package desiccants can be avoided with the help of extractive drying, since orange powders at moisture contents below 1.0% can be achieved.

LIST OF CITRUS PUBLICATIONS AND PATENTS

(September 1, 1960 - August 31, 1961)

UNITED STATES DEPARTMENT OF AGRICULTURE
AGRICULTURAL RESEARCH SERVICE

SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

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THE DETERMINATION OF SOLUBLE SOLIDS IN CITRUS JUICES. I. THE EFFECT OF NON-SUCROSE COMPONENTS ON REFRACTOMETER VALUES
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(All patents are assigned to U. S. Government as represented by the Secretary of Agriculture)

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DEHYDRATION OF FRUIT AND VEGETABLE JUICES
A. I. Morgan, Jr. and L. F. Ginnette
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PROCESS OF DEHYDRATING FRUIT AND VEGETABLE JUICES
A. I. Morgan, Jr., J. M. Randall, and R. P. Graham
U. S. Patent No. 2,967,109, Jan 3, 1961

1/ Patents can be obtained only by purchase from the U. S. Patent Office, Washington 25, D. C., for 25 cents each.

Reprints of the above - listed publications are available and may be obtained without cost by addressing request to the respective laboratories.